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N-Methyl-2-Pyrrolidone for industrial use

工业用 N-甲基-2-吡咯烷酮

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Standardization Administration of the People's Republic of China.

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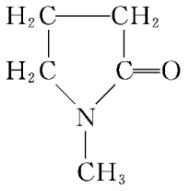
N-Methyl-2-Pyrrolidone for industrial use

1 Scope

This Standard specifies the requirements, test methods, inspection rules, as well as marking, packaging, transportation and storage of N-methyl-2-pyrrolidone for industrial use.

This Standard applies to N-methyl-2-pyrrolidone synthesized from γ-butyrolactone and methylamine.

Chemical formula:C_5H_9ON

Structural formula: 

Relative molecular mass: 99.13 (according to the 2007 international relative atomic mass)

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 325.1, *Zinc-coated steel wire ropes for high-pressure rubber hose*

GB/T 601, *Chemical reagent -- Preparations of reference titration solutions*

GB/T 3143, *Color determination method of liquid chemicals (Hazen unit - Platinum-cobalt scale)*

GB/T 6283-2008, *Chemical products -- Determination of water Karl-Fischer method (general method)* (ISO 760:1978, NEQ)

GB/T 6488, *Liquid chemicals -- Determination of refractive index at 20°C*

GB/T 6678, *General principles for sampling chemical products*

GB/T 6680, *General rules for sampling liquid chemical products*

GB/T 6682, *Water for analytical laboratory use -- Specification and test methods* (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 8170, *Rules of rounding off for numerical values and expression and judgement of limiting values*

GB/T 9722, *Chemical reagent -- General rules for the gas chromatography*

GB/T 9724-2007, *Chemical reagent -- General rule for the determination of pH* (ISO 6353-1:1982, NEQ)

GB/T 9725-2007, *Chemical reagent -- General rule for potentiometric titration* (ISO 6353-1:1982, NEQ)

3 Requirements

3.1 Appearance

It is a colorless or slightly yellow transparent liquid at room temperature.

3.2 Requirements

N-methyl-2-pyrrolidone for industrial use shall meet the technical requirements shown in Table 1.

4 Test methods

4.1 Warning

Some test procedures specified in the test method may lead to dangerous situations. Operators shall take appropriate safety and protective measures.

4.2 General provisions

Unless otherwise specified, only analytically pure reagents and grade 3 water as

specified in GB/T 6682 are used in the analyses.

Unless otherwise specified, the standard titration solutions used in the analysis shall be prepared in accordance with the provisions of GB/T 601.

4.3 Appearance

Take an appropriate amount of laboratory sample into a colorimetric tube. Observe visually under natural light.

4.4 Determination of N-methyl-2-pyrrolidone content

4.4.1 Method summary

Gas chromatography is used. Under selected chromatographic conditions, the specimen is vaporized and passed through a capillary column to separate the components. Detection is by flame ionization detection (FID). Quantification is performed using the corrected area normalization method. After subtracting water and total amines, the N-methyl-2-pyrrolidone content is calculated.

4.4.2 Reagents

4.4.2.1 Nitrogen: volume fraction ≥ 99.995%.

4.4.2.2 Hydrogen: volume fraction ≥ 99.99%.

4.4.2.3 Air: purified with activated carbon and blue silica gel, then dried.

4.4.2.4 N-methyl-2-pyrrolidone: chromatographically pure.

4.4.2.5 γ-Butyrolactone: chromatographically pure.

4.4.2.6 1,4-Butanediol: chromatographically pure.

4.4.2.7 2-Pyrrolidone: chromatographically pure.

4.4.3 Instruments

4.4.3.1 Gas chromatograph: equipped with a flame ionization detector (FID). The sensitivity and stability of the instrument shall comply with the requirements of GB/T 9722. The linear range shall meet analytical requirements.

4.4.3.2 Data processing system: chromatography data processor or chromatography workstation.

4.4.3.3 Sample injector: 1.0 μL micro syringe or automatic sample injector.

4.4.4 Chromatographic analysis conditions

4.5 Determination of moisture

The determination shall be carried out in accordance with the provisions of Chapter 8 of GB/T 6283-2008.

The arithmetic mean of the two parallel determination results shall be reported as the result. The absolute difference between the two parallel determination results shall not exceed 0.003%.

4.6 Determination of chromaticity

The determination shall be carried out in accordance with the provisions of GB/T 3143.

4.7 Determination of refractive index

The determination shall be carried out in accordance with the provisions of GB/T 6488.

4.8 Determination of total amine content

4.8.1 Method summary

Dissolve the sample in isopropyl alcohol. Titrate the alkaline substances in the specimen with a standard hydrochloric acid solution. Use a potentiometer or pH meter to indicate the titration endpoint. The total amine content is calculated as monomethylamine.

4.8.2 Reagents

4.8.2.1 Isopropyl alcohol.

4.8.2.2 Standard hydrochloric acid titration solution: c(HCl) = 0.02 mol/L.

4.8.3 Instruments

4.8.3.1 Potentiometer: the accuracy of the potentiometer is ±0.01 mV.

4.8.3.2 Acidity meter: the accuracy of the acidity meter is pH ± 0.01, with temperature compensation function.

4.8.4 Analysis steps

Weigh 65 g of laboratory sample to the nearest 0.01 g. Place in a 250 mL beaker. Dissolve in 100 mL of isopropyl alcohol. Shake well. Perform the following determination according to Chapter 6 of GB/T 9725-2007.

4.8.5 Result calculation

The total amine content is expressed as the mass fraction w_2 of monomethylamine (CH_3NH_2), expressed in %, and calculated according to formula (3):

$$w_2 = \frac{VcM}{m} \times 100 \qquad \cdots\cdots(3)$$

Where,

V - the volume of the standard hydrochloric acid titrant (4.8.2.3) consumed by the test material, in milliliters (mL);

c - the exact concentration of the standard hydrochloric acid titrant, in moles per liter (mol/L);

m - the mass of the test material, in grams (g);

M - the molar mass of monomethylamine (CH_3NH_2), in grams per mole (g/mol) (M = 31.06).

The arithmetic mean of the two parallel determination results shall be reported as the result. The absolute difference between the two parallel determination results shall not exceed 0.001%.

4.9 Determination of pH

Use a pipette to transfer 10.0 mL of the sample to a 100 mL volumetric flask. Dilute to the mark with carbon dioxide-free water. Shake well and transfer to a beaker. The following steps shall be performed in accordance with Chapter 6 of GB/T 9724-2007.

5 Inspection rules

5.1 All items specified in 3.1 and 3.2 Table 1 of Chapter 3 are exit-factory inspection items. Exit-factory inspection shall be carried out on each batch.

5.2 N-methyl-2-pyrrolidone for industrial use is shipped in batches based on the quantity of one storage tank or tank truck (ship), or products of equivalent mass.

5.3 The sampling method and number of sampling units for N-methyl-2-pyrrolidone for industrial use shall comply with the requirements of GB/T 6678 and GB/T 6680. The sampling volume shall be no less than 400 mL. Divide the sample into two clean, dry glass bottles and seal them tightly. Label each bottle with the product name, batch number or production date, sampling date, sampling location, and the name of the sampler. One bottle shall be used for testing, and the other bottle shall be retained for future reference.

5.4 N-methyl-2-pyrrolidone for industrial use shall be inspected by the manufacturer's quality supervision and inspection department. The manufacturer must ensure that each batch of product meets the requirements of this Standard. Each batch of product must

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